

Issues involving infrared detector material systems

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Objectives and Status—The original objectives of this project are two folded. One objective is to develop textured template for growth of **epitaxial** thin film ferroelectric (TFFE) IR detectors on polyimide coated Si. The commercial TFFE has a polycrystalline structure since it is directly grown on non-textured polyimide coating. This results in degraded device performance. The same problem also occurs widely to many other device applications. To achieve epitaxial growth of the devices on non-textured surface, an ultra-thin textured template, with high transparency to infrared photons in the infrared detector applications, must be developed on non-textured surface. To resolve this issue, we have developed a unique approach in this project to generate ultra-thin textured MgO buffers on amorphous substrates using ITEX technique (stand for ion beam texturing). This topic has been the main focus of this project and many exciting results have been obtained. The other topic is to improve the performance of Hg-Cd-Te by engineering substrate surface at nano-scales aiming at reducing the defect densities such as dislocations in the device. The main approach is to prepare surfaces that have alternating areas of perfect lattice match—area 1 or no match (amorphous, for example)—area 2 to Hg-Cd-Te. Each area has a dimension on the order of few tens of nanometers. It is anticipated that Hg-Cd-Te will only nucleate in area 1, not area 2. If the Hg-Cd-Te domains grown in area 1 can overlay on the area 2, the strain induced from lattice mismatch between substrate and Hg-Cd-Te layer may be minimized. We have successfully achieved the surface nano-engineering using electron-beam lithography (EBL). The dimension of the engineered surface area is, however, small typically on the order of few hundred micrometers. This topic was not pursued after one year experiment, due to limitation of characterization capability on small sample spot on our ARL collaborator side.

This project also involved a strong educational component. A graduate student, Mr. Ronald Vallejo and an undergraduate student, Alan Dibos were supported during the almost the whole project period. Mr. Javier Baca, another PhD student was also supported for one year period at the beginning of the project. Mr. Vallejo had been working on generating textured template on Si, glass, and polyimide coated Si substrates using ITEX technique. An advanced high vacuum ITEX system was designed and constructed during the first two years of the project. In addition, Mr. Vallejo also spent some efforts in growth of TFFE devices. He has completed his PhD degree recently. Mr. Dibos is a Physics senior and he has been working on nano-engineering of Si substrate surface using electron beam lithography (EBL). He graduated in the spring of 2006 and is attending graduate school at the Harvard University. We have requested 12 month no-cost extension of the project, which has been approved, to support Mr. Vallejo to complete his Ph.D. thesis work by the summer of 2006. Mr. Baca made considerable contribution in design and assembling our ITEX

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system. He is currently working his PhD thesis on microstructure characterization of the samples including ones made by Dr. Vallejo at the Electron Microscopy facility of the US Air Force research lab in Dayton, Ohio.

Scientific progress and accomplishments—The major focus of the reporting period is on development of the ITEX process and on investigation of the texture developing mechanism of MgO in the ITEX process. The ITEX MgO templates provide a unique solution to the generic problem of epitaxy devices on non-textured substrates or the ones with large lattice mismatch with the device materials. Exciting results have been achieved including achievement of 10 nm thick MgO epitaxial templates that are completely transparent on un-buffered glass, Si, and polyimide coated Si substrates. We then proceeded in growing TFFE on top of the MgO templates and improved device performance has been obtained. In addition, we have also developed bi-crystal templates. The technique is unique and innovative and may have a promising impact on many device applications. We have also explored the second topic of surface engineering for Hg-Cd-Te (MCT) infrared detectors in the first year of the project and some interesting results have obtained. The details of the results obtained during the project period are summarized in the following.

(1) Development of textured template on non-textured surface using ITEX technique

(a) **Design and construction** of the ITEX system—were completed within 12 months after the project began. It took about six months for full calibration of all the components and in-situ measurements. Two e-beam evaporation sources were installed with one has a single pocket and the other, four pockets for sequential growth. The ITEX system is also equipped with a 3 cm Kauffman ion source mounted at 45 degree with respect to the normal of the sample. The ion beam current is in situ monitored using a Farady cup and the film growth rate, using a quartz oscillator. A high pressure RHEED system was also installed for *in situ* monitoring the layer-by-layer epi-growth. Figure 1 depicts the schematic and picture of this ITEX system.

(b) **Development of textured MgO template** has been carried out successfully using the ITEX system. A process for growth of 10 nm thick textured MgO templates on un-buffered commercial Si and glass substrates has been developed, which differs from previously reported work in terms of a stack of buffer layers must be laid on substrates before the MgO template growth. Interestingly, we have found that pre-exposure of the substrate to ion beam for certain period ranging from several minutes to tens of minutes can prepare the substrates for MgO epi growth and the texture quality in this MgO template is comparable to the best reported previously (see Figure 2). Since a 10 nm thickness is adequate for texture to form in ITEX MgO, this work is important for many devices that require thin epi-templates. Additional buffers can significantly thicken the epi-template and cause degradation of the device performance. This work is also important to the basic science of the ion beam assisted texturing process because its mechanism, such as the necessary and adequate conditions for the epi MgO nucleation to occur remains a mystery. From our study, we have found the ion beam pre-exposure smoothens the surface of the substrates and the ITEX MgO can be obtained when the surface roughness is on the order of 1-2 nm or better. We have also discovered that while surface roughness is a necessary condition, it is not the adequate one. Additional surface activation generated by the ion beam pre-exposure (or buffer layers in previous work) is another critical condition for textured MgO to nucleate.

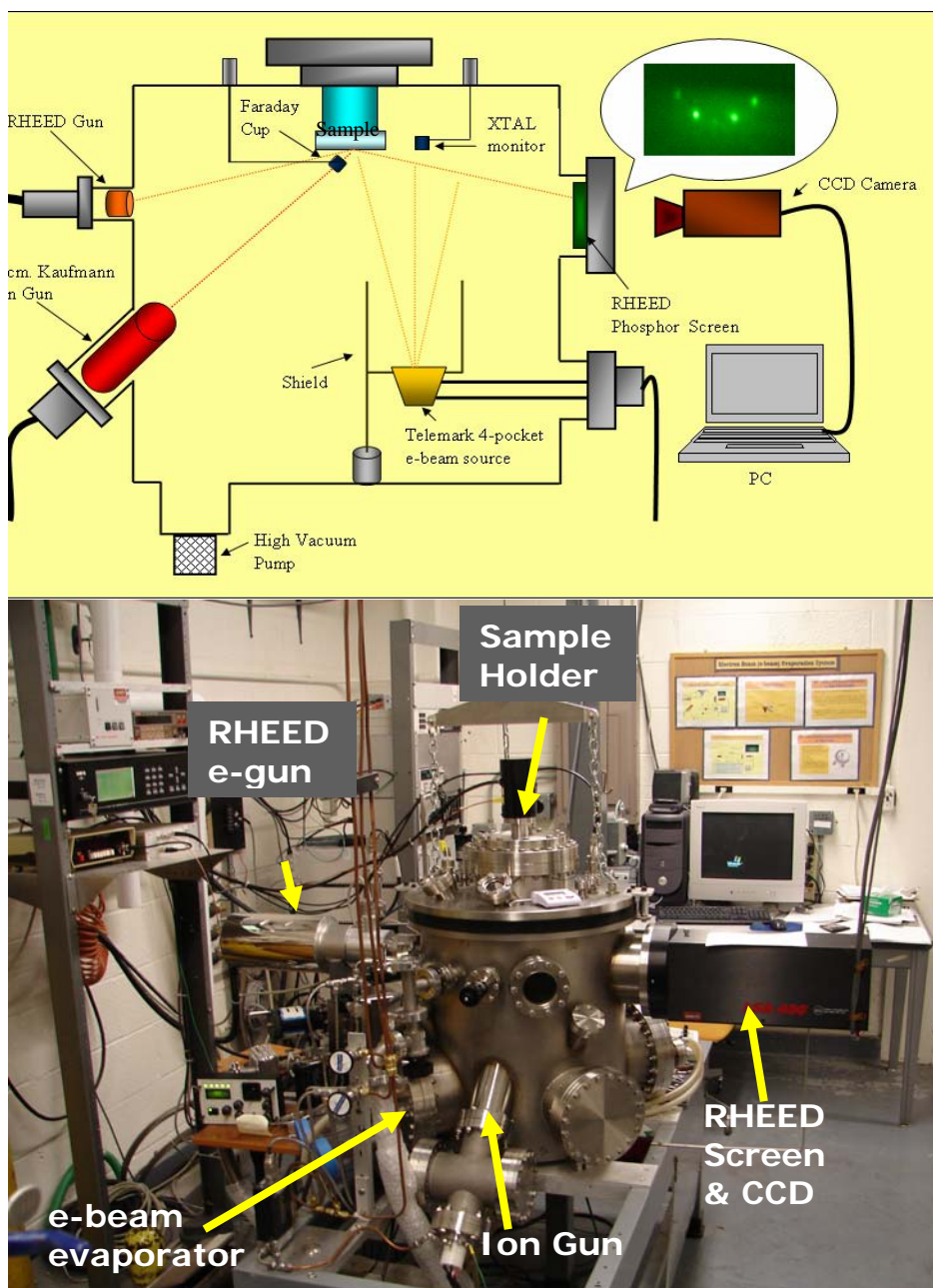


Figure 1. Schematic (upper panel) and picture (lower panel) of the high vacuum ion beam texturing system (ITEX system).

(c) **ITEX MgO template on polyimide coated Si** was one of our research focuses, aiming at development of suspended TEEE infrared detectors of epi-structure and therefore optimized performance. It should be noted that the polymer surface presented a challenge to the ITEX process due to the unfavorable ion beam interaction with polymer. It has been found that Ar ions interact with polyimide surface physically, generating large pits, leading to roughened surface morphology which prevents textured MgO to nucleate (see Figure 3). On the other hand, O ions that exist even in vacuum interact with polyimide surface physically and chemically, sputtering off most of C and N, creating active sites for O attachment, and weakening the carbon to carbon bond. This results in degradation of polyimide (chains broken) surface integrity. Direct application of the process

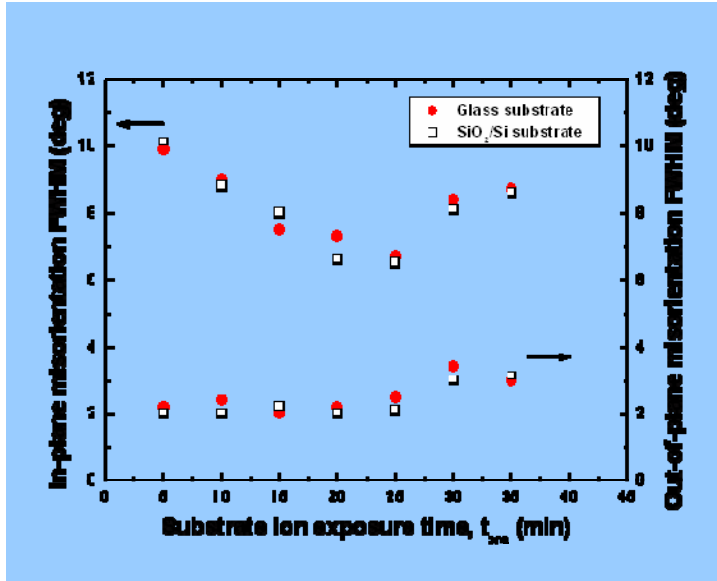


Figure 2. XRD in-plane and out-of-plane mis-orientation as function of ion-beam pre-exposure time. Pre-exposure has two effects: smoothening the surface morphology and activating the surface chemical bonding.

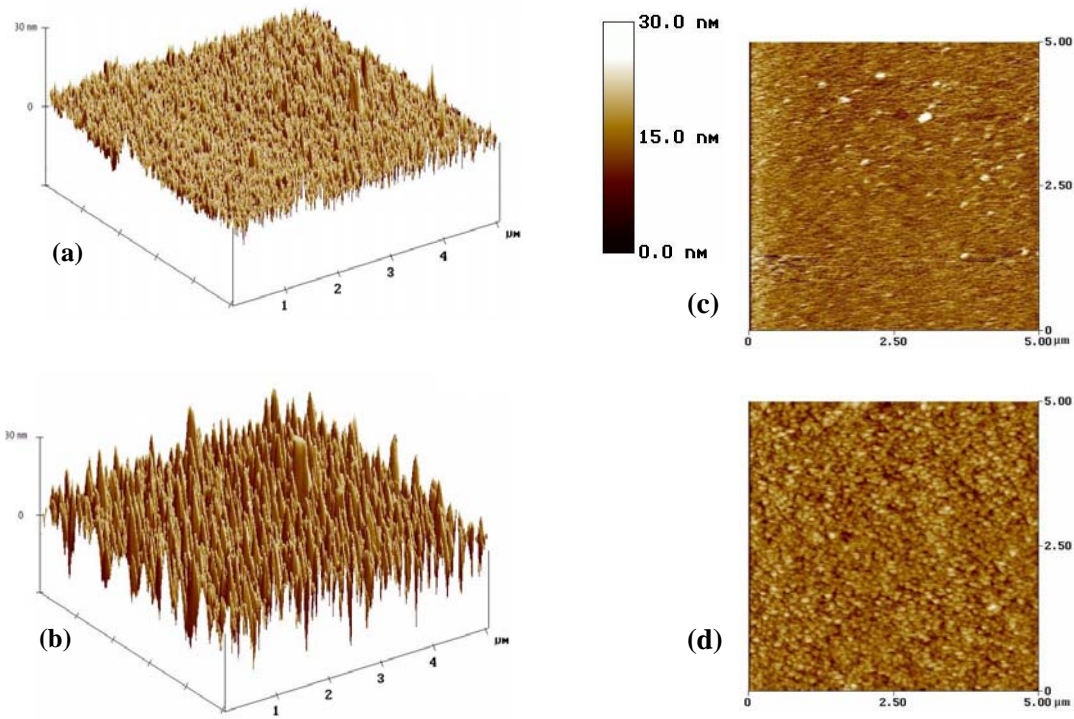


Figure 3. AFM images of (a) original polyimide surface of surface roughness $R_a \sim 1-2\text{nm}$; (b) after 10 minutes pre-ion beam exposure with $R_a \sim 5-7\text{nm}$; (c) the original surface covered with 25 nm thick Y_2O_3 with $R_a \sim 1-2\text{nm}$; and (d) after 20-22 nm of the Y_2O_3 being removed with $R_a \sim 1-2\text{nm}$. Good quality ITEX MgO has been obtained on this surface.

developed for glass and Si substrates failed to generate epi MgO on polyimide due to the above mentioned reasons. To solve this problem, we have introduced a 25 nm thick Y_2O_3 buffer on polyimide and then remove 80-90% of it using ion beam pre-exposure before ITEX MgO growth. Good quality epi MgO templates have been obtained (see Figure 3). The TFFE devices grown atop show much improved crystalline texture and performance.

(d) **Bi-crystal arrays** have also been developed using ITEX technique. Traditionally, bi-crystals are made by fusing two pieces of single crystals together at extremely high temperatures. The typical dimension of each crystal is on the order of millimeters or larger. The method itself presents a fundamental limit on going smaller dimension and complicate network needed for device applications. Motivated by this, we have developed a process combining ITEX and lithography so that different orientation templates can be grown at specified location and dimension (US patent pending). Briefly, small dimension textured templates are laid on substrates with lithographically defined areas. The liftoff process is then applied to expose other areas for second template growth of different crystal orientation. Figure 4 depicts a bi-crystal array generated in this process. The squares are 45 degree rotated with respect to the rest of the sample. It should be realized that this method has no limitation on the dimension (controlled by lithography) and orientation (control by ITEX parameters) of each crystals in the array.

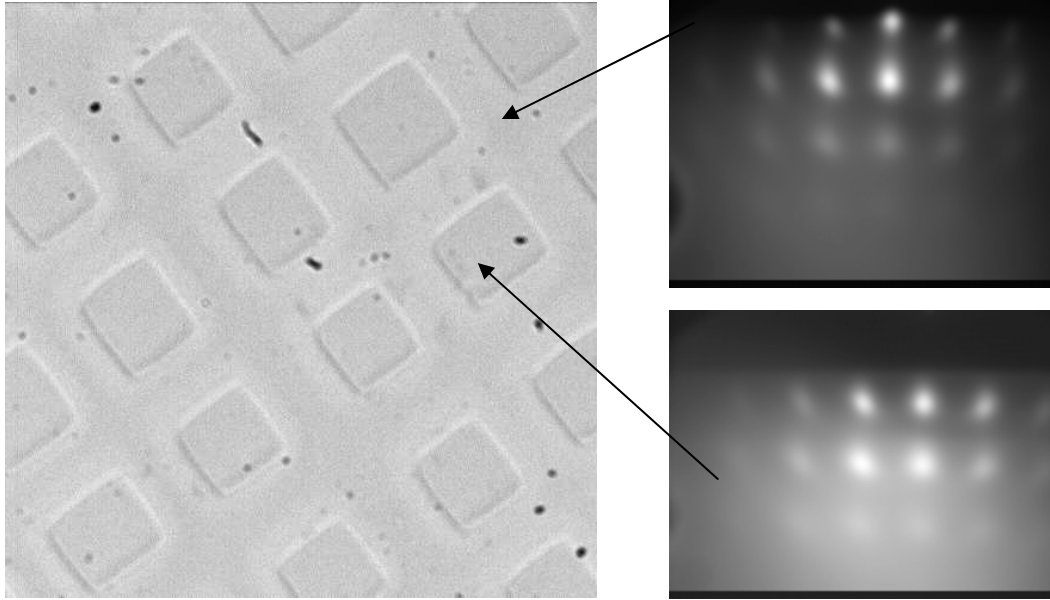


Figure 4. Bi-crystal array made using ITEX method in combination of photolithography. The squares have a dimension of $10 \times 10 \mu\text{m}^2$ and their in-plane structure rotates 45 degree with respect to the background as shown in RHEED patterns on the right.

(2) Development of Nano-structured templates on Si for Hg-Cd-Te

This part of research explored new ideas of engineered templates for MCT devices. One idea we have studied is to generate surfaces that have alternating areas of perfect lattice match—area 1 or no match (amorphous, for example)—area 2 to MCT. It is anticipated that MCT will only nucleate in

area 1, not area 2. If the MCT nucleation in area 1 can overlay on the area 2, the strain caused by lattice mismatch between the substrate and MCT may be minimized (see Figure 5). The experimental procedure was as follows: (1) the (211) SiO₂ substrates covered with CdTe buffer was covered with a thin (20 nm thick) SiO_x layer using e-beam evaporation; and (2) electron beam lithography was then performed to generate stripes (area 2) on the PMMA (e-beam resist). The width of the strip is ideally 20 nm or smaller and the separation between strips is in the range of 200-500 nm. Figure 5 includes a scanning electron microscopy (SEM) picture of ten parallel lines written using e-beam lithography. The line width is around 90 nanometers and the inter-line distance is around 1 micrometer. One of the obstacles that has held this project is to write the pattern in large area using EBL within reasonable time. Theoretically, it can be done using long beam writing time at a high cost.

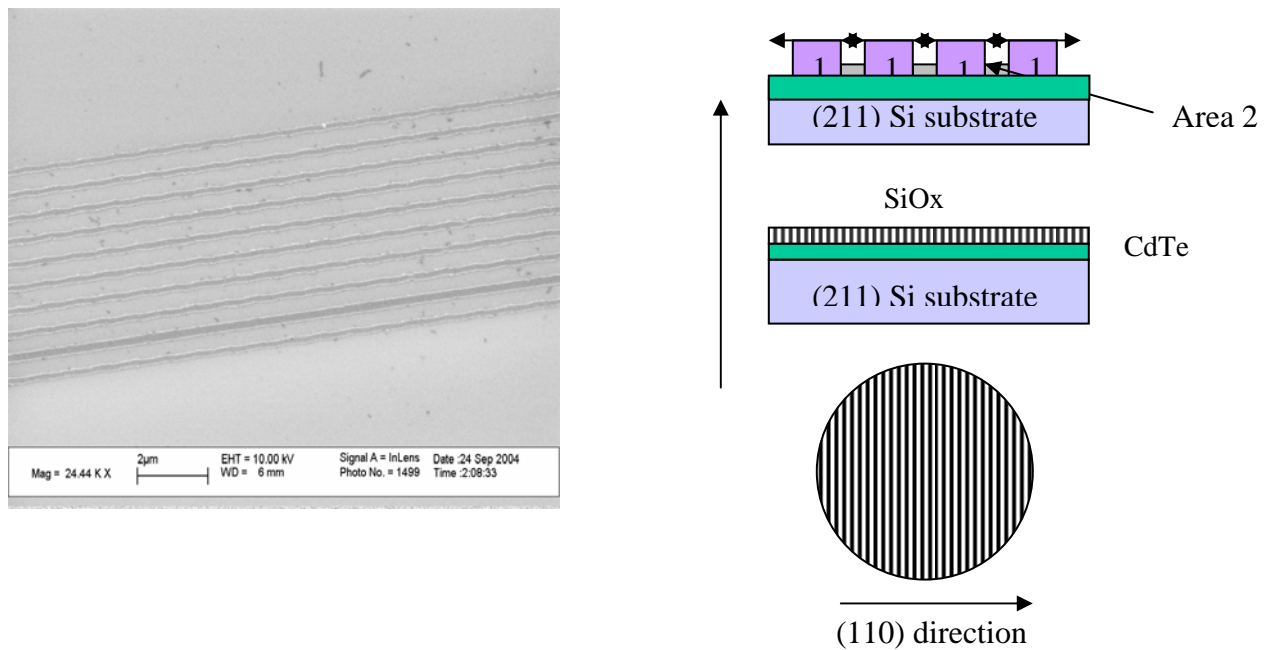


Figure 5 (right side) shows the top view (bottom), side view (middle) of the nano-engineered Si substrates and the side view of MCT layer grown on this substrate (top). Left side shows SEM image of modulated SiO_x layer generated using E-beam lithography.

Publications:

1. (US patent pending) J.Z. Wu and R. Vallejo, "Bi-crystal network templates and method of fabrication thereof".
2. R. Vallejo and J.Z. Wu, "Development of MgO texture templates on polymers", preprint
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4. R. Vallejo and J.Z. Wu, "Ion Beam Assisted Deposition of textured Magnesium Oxide templates on un-buffered glass and silicon substrates", J. Mat. Res. **21**, 194 (Jan. 2006)
5. R. Vallejo, S.H. Yun, J.Z. Wu, M. Tidrow, H. Braaten, C. Hansen, and P. Arendt, "Effect of in-plane and out-of-plane misorientation on the ferroelectric properties of thin film ferroelectric

- PZT infrared sensors on Si substrates”, Proceeding of SPIE Aerosense Conf. Orlando, April 20-24, 2003.
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